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A NEW DERIVATIVE OF DIAZO-AMIDO-BENZENE.

(2, 2′ dibrom, 4, 4′ dimethyl diazo amido-benzene.)

By EDWARD BARTOW and H. C. ALLEN, University of Kansas, Lawrence.

Read before the Academy, at Topeka, December 31, 1904.

M-BROMTOLUENE may be prepared from *p*-acettoluid by bromination, elimination of the acetyl group, and submitting the amine thus formed to the Griess reaction,[1] thus replacing the amido group with hydrogen.

In following out the Griess reaction for the diazotizing of *m*-brom-*p*-toluidine by means of nitrous acid, we obtained well-defined bright yellow, needle-like crystals. Wroblevsky,[2] who prepared *m*-bromtoluene by the above-mentioned series of reactions, warms the alcoholic solution after diazotizing to effect reduction and makes no mention of an intermediate product. Sellards[3] mentions a crystal broth which is formed before the reduction takes place.

Our method of procedure was as follows: *P*-acettoluid was suspended in water and brominayed according to Wroblevsky[2] by adding bromine with occasional shaking, but without particular care to cool the reaction mixture. Under these circumstances *m*-brom-*p*-acettoluid is formed.

The acetyl group, which serves to protect the amine, is then removed by saponifying with either NaOH or KOH. Wroblevsky[2] claims that only KOH will act, while Grete[4] and Sellards[3] find that NaOH does equally well. Our experience was that either would react, but that the NaOH required longer heating, and also heating on the sand-bath. The *m*-brom-*p*-toluidine was then distilled with steam, dried with calcium chloride and diazotized by leading nitrous acid into a solution in absolute alcohol. The nitrous acid is best prepared by using arsenic trioxide having a specific gravity of 1.3 to 1.35. The nitrous acid was conducted through a reflux condenser, cooled by ice-water, and a Woulff's flask, surrounded by a freezing mixture, into the alcoholic solution. The condenser and flask serve as traps for nitric acid as well as for cooling the nitrous acid. The gas was absorbed until near the end of the reaction, and at the same time the bright yellow crystals mentioned above were formed.

If any acid is present in the alcoholic solution, the substance formed will be reduced to *m*-bromtoluene. Ten drops of sulfuric acid in ten grams of the substance completely reduced it in thirty minutes. When no acids are present, however, the crystals are very

1. A, 121-258. 2. A, 168-154. 3. Dissertation, U. of K., 1904. 4. A, 177-232.

stable and can be isolated. They are soluble in ether, but only slightly so in alcohol, and are purified by washing with alcohol and recrystallizing from a mixture of ether and alcohol.

A determination for bromine gave the following results:

0.2151 grams of the substance gave 0.2100 grams of AgBr.

Calculated for $C_{14}H_{13}N_3Br_2$.	Found.
41.77%	41.58%

A molecular weight determination, by lowering of the freezing-point of benzene, gave the following:

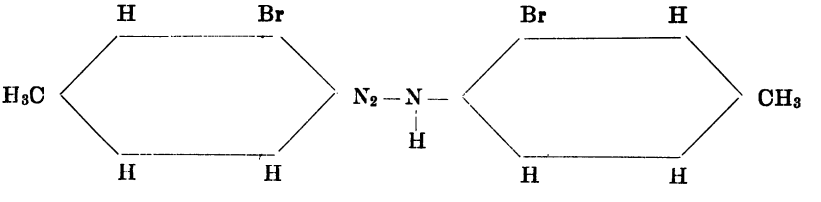
I. 0.2435 grams caused a depression of 0.223 degrees in the freezing-point of 14.13 grams of benzene.

II. 0.4459 grams caused a depression of 0.42 degrees in the freezing-point of 14.13 grams of benzene.

	Calculated for $C_{14}H_{13}N_3Br_2$.	Found.
I.....	382.13	378.66
II.....	.....	368.16

If, as claimed by Griess, aniline treated in this way gives diazo-amido-benzene, then our reaction may be represented as follows:

I. $C_6H_3CH_3BrNH_2 + HONO = C_6H_3CH_3BrN=NOH + H_2O.$
m-brom-*p*-toluidine nitrous acid = *m*-brom-*p*-diazotoluene.

II. $C_6H_3CH_3BrN=NOH + C_6H_3CH_3BrNH_2 =$
$C_6H_3CH_3BrN=N-NH-C_6H_3CH_3Br + H_2O.$
4, 4′ dimethyl 2, 2′ dibrom diazo-amido-benzene.

The substance may be graphically represented: